NATIONAL BUREAU OF STANDARDS REPORT

7836

CURRENT NBS EFFORTS TOWARD DEVELOPMENT
OF THERMAL CONDUCTIVITY REFERENCE STANDARDS

Complementary Report
March 1963

by

D. R. Flynn

to the

Bureau of Ships
Department of the Navy
Washington, D. C.

U.S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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Heat Transfer Section
Building Research Division

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Complementary Report
March 1963

NOT FOR PUBLICATION
OR FOR REFERENCE

by D. R. Flynn

ABSTRACT

This report discusses the efforts which are currently being made at the National Bureau of Standards in the direction of developing suitable thermal conductivity reference standards:

1. Armco Iron - NBS data are presented for the thermal conductivity of Armco iron over the temperature range from -160° to 640°C. Below 400°C the NBS results are within 1 percent of those obtained by R. W. Powell of the National Physical Laboratory on a specimen from the same lot.

2. Lead - A comparison is given of published values of the thermal conductivity of lead over the temperature range from -250° to 300°C. This review was made as a preliminary to possible determination of the thermal conductivity of NBS freezing point lead.

3. Inconel 702 - Thermal conductivity results obtained by four independent methods at NBS are given over the temperature range from -150° to 1200°C. For samples of this alloy having similar thermal histories, the smoothed results are within about 1 percent of a smooth curve.

4. Pyroceram Code 9606 - Measurements have been made over the temperature range 200° to 1000°C by one method; the thermal conductivity values obtained were 0.035 w/cm C at 200°C and 0,028 w/cm C at 1000°C. Additional measurements by other methods are in progress.

5. Pyrex Glass (Code 7740) - A brief survey of published data on the thermal conductivity of Pyrex over the temperature range 0° to 500°C is given. Some unpublished NBS data are included. Additional NBS measurements are planned.

1. INTRODUCTION

This report is a slightly modified version of a paper given by the author at the Second Conference on Thermal Conductivity held at the National Research Council of Canada, Ottawa, Canada, on October 10-12, 1962. It is

being made available as an NBS report in the belief that the contents may be of general interest. This report discusses the efforts which are currently being made at NBS with the aim of developing suitable thermal conductivity reference standards. Many of the considerations involved in the selection and use of reference standards are discussed in a separate report [1][1/].

In the discussion below, thermal conductivity data are presented which have not been formally published. All such data are subject to review, editorially and otherwise, prior to formal publication, and their preliminary status should be recognized.

2. ARMCO IRON

Powell [2] has recently reviewed the numerous published data now available on the thermal conductivity of Armco iron and derived "most probable values," which are estimated to be good within ±2 percent from 0° to 600°C, and within about ±5 percent at 1000°C. The range of experimental values in the literature, however, is about 7 percent between 0° and 600°C, and increases to almost 30 percent at 1000°C. In an attempt to resolve this discordance of experimental results, C. F. Lucks of the Battelle Memorial Institute proposed a round-robin of thermal conductivity measurements on Armco iron. The Battelle Memorial Institute obtained samples of Armco iron in the form of 1-inch diameter rods, annealed them for half an hour at 850°C, and distributed samples to: National Physical Laboratory, Teddington, England; National Research Council, Ottawa, Canada; and National Bureau of Standards, Washington, D. C., U.S.A., and later to a number of other laboratories. R. W. Powell, et al. have reported [3] the NPL measurements over the temperature range from -200° to 1000°C; M. J. Laubitz has reported [4] the NRC measurements from 30° to 1000°C.

The sample of Armco iron supplied to NBS by Battelle was in the form of a round bar, nominally 1 inch in diameter and 39 inches in length. The ends of this sample were stamped No. 3 and No. 4, the No. 3 end being immediately adjacent to the sample sent to the National Physical Laboratory, and the No. 4 end adjacent to the sample sent to the National Research Council. This Armco iron was stated to have been obtained from the American Rolling Mill Company, Hamilton, Ohio. The ladle analysis in weight percent, as given by the supplier, is as follows: C 0.02, Mn 0.030, P 0.006, S 0.023, Si 0.004, Cu 0.083, Fe (by difference) 99.834. Powell [3] reported that an NPL analysis showed no significant variation from the analysis given above, other than the presence of 0.083 percent Ni.

1/ Figures in brackets indicate the literature references at the end of this report.

TABLE 1

THERMAL CONDUCTIVITY* OF BATTELLE ARMCO IRON

Temperature °C	NBS k	NPL k	NPL ,%	"Most Probable" k	"Most Probable" ,%
-160	0.887	0.893**	0.7	-----	----
-150	.873	.879	0.7	-----	----
-100	.815	.820	0.6	-----	----
-50	.775	.778	0.4	-----	----
0	.742	.747	0.7	0.749	0.9
50	.712	.719	1.0	-----	----
100	.682	.686	0.6	.672	-1.5
150	.652	.654	0.3	-----	----
200	.620	.621	0.2	.608	-1.9
250	.587	.588	0.2	-----	----
300	.554	.555	0.2	.548	-1.1
350	.523	.525	0.4	-----	----
400	.495	.492	-0.6	.487	-1.6
450	.469	.460	-1.9	-----	----
500	.443	.430	-2.9	.433	-2.3
550	.417	.405	-2.9	-----	----
600	.391	.382	-2.3	.389	-0.5
640	.371	.364**	-1.9	-----	----

* Watt/cm deg C

** Interpolated

The NBS Heat Transfer Section has completed measurements of the thermal conductivity of the Battelle Armco sample over the temperature range from -160° to 640°C. These measurements were made in the NBS metals apparatus, which has been described by Watson and Robinson [5]. In brief, the measurement was made by determining the electrical power input to a heater in one end of a specimen 37.0 cm long and 2.386 cm in diameter, which was cooled at the other end by circulating water or liquid nitrogen. Temperatures were measured by means of thermocouples at seven locations, 3.51 cm apart, along the central portion of the bar, thus permitting the calculation of six thermal conductivity values, each at a different mean temperature, for each thermal equilibrium. A guard cylinder, concentric with the specimen, was used to minimize heat exchanges between the specimen and the surrounding insulation, and corrections were made for such heat exchanges.

Twelve data points were obtained in the temperature range from -160° to 192°C in the NBS low-temperature metals apparatus [5], using 0.40 mm diameter chromel:alumel thermocouples, diatomaceous earth insulation in air, and liquid nitrogen coolant. Eighteen data points were obtained in the temperature range from 90° to 508°C in the NBS high-temperature metals apparatus [6], using 0.38 mm diameter platinum-10% rhodium:platinum thermocouples, fine powdered alumina insulation flushed with 4 liters/hr of dry nitrogen gas, and 40°C water coolant. Eighteen additional data points were obtained in the temperature range from 125° to 650°C in the high temperature apparatus, using a new heater, the same thermocouples, the same alumina insulation evacuated to about 15 Torr, and 40°C water coolant.

Table 1 presents the smoothed NBS results at 50 deg C intervals. Powell's data on the Battelle Armco are given in the third column with percent departures (NBS = 100) in the fourth column. Powell's "most probable values" are given in the fifth column, with percent departures (NBS = 100) in the sixth column.

The solid line in the lower drawing of Figure 1 represents the NBS data given in Table 1; the broken line represents Powell's results for Battelle Armco (Table 1, column 3). The upper drawing in Figure 1 shows the percent departures of the NPL data from those obtained at NBS (Table 1, column 4).

The NBS results have not yet been corrected for the effects of longitudinal heat flow in the surrounding powder insulation. This adjustment may result in lowering the NBS values by about one-third percent at 0°C and about one percent at 600°C. Aside from this correction, uncertainties in the results given are estimated to be about one percent.

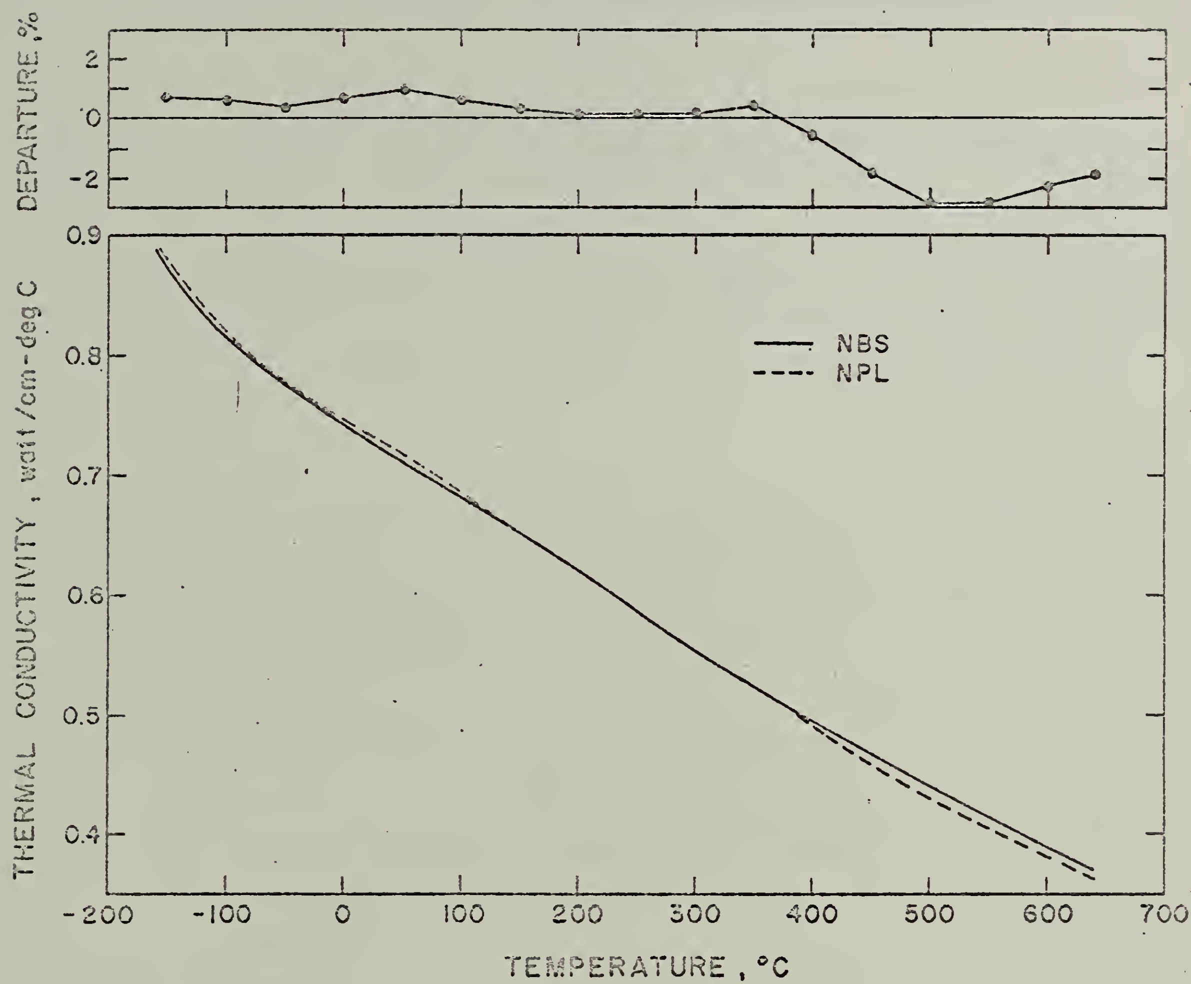

FIGURE 1. THERMAL CONDUCTIVITY OF ARMCO IRON (SAMPLE SUPPLIED BY BATTELLE MEMORIAL INSTITUTE). THE UPPER DRAWING SHOWS THE DEPARTURES OF THE NPL RESULTS FROM THOSE OBTAINED AT NBS

Electrical resistivity measurements have been made from liquid nitrogen temperatures to room temperature, and are in good agreement with the results of Powell [3] over this range. High temperature electrical resistivity measurements on a sample of Battelle Armco will be made soon.

3. LEAD

Van Dusen and Shelton [7] selected lead as the primary standard for their comparative method of thermal conductivity measurement. At the time, they felt that the thermal conductivity of lead was known within 3 percent or better at the ice point, and assumed the value to be 0.352 watt/cm deg C at 0°C, as given in the International Critical Tables. They determined the temperature coefficient (to 300°C) as 0.057 percent decrease per degree C. Since that time, several investigators [8,9,10,11,12] have either used lead as a primary standard in a comparative apparatus, or have checked their equipment using lead.

In view of the continued use of lead as a thermal conductivity reference standard, it was decided to survey the literature to determine the need for a new absolute measurement of the thermal conductivity of lead. The curve in the lower drawing in Figure 2 shows values we have deduced from the literature for the thermal conductivity of lead as a function of temperature. The values plotted are listed in Table 2.

TABLE 2

DEDUCED THERMAL CONDUCTIVITY OF LEAD

Temperature	k
-250°C	0.484 w/cm C
-200	.402
-100	.368
0	.352
100	.336
200	.320
300	.305

These values were obtained by fairing a curve through the available literature data over the temperature range from -250° to 300°C. The percent

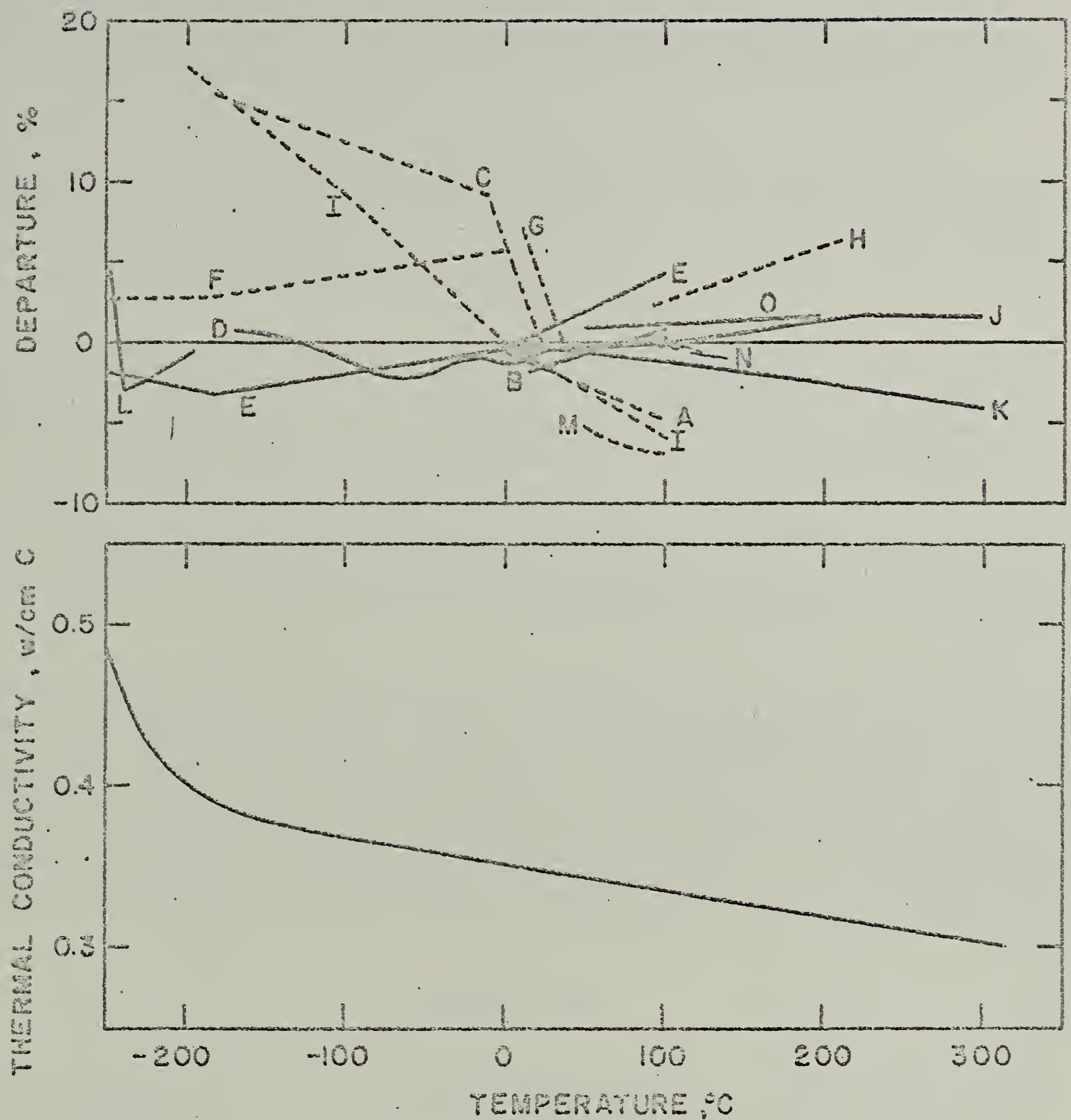

FIGURE 2. DEDUCED VALUES FOR THE THERMAL CONDUCTIVITY OF LEAD. THE UPPER DRAWING SHOWS DEPARTURES OF DATA OF VARIOUS INVESTIGATORS FROM THE DEDUCED VALUES

A. Lorenz (13)
B. Jaeger & Diesselhorst (14)
C. Macchia (15)
D. Lees (16)
E. Meissner (17)
F. Schott (18)
G. Peczalski (19)
H. King (20)
I. Bidwell & Lewis (22)
J. Konno (21)
K. Van Dusen & Shelton (7)
L. Bremmer & de Haas (23)
M. Scholes (8)
N. Weeks & Seifert (9)
O. Francl & Kingery (10)

departures of the individual investigations [13,14,15,16,17,18,19,20,21,22, 7,23,8,9,10] from this smooth curve are shown in the upper drawing in Figure 2. The construction of the smooth curve shown in the lower drawing was somewhat arbitrary, in that the investigations indicated by solid lines in the upper drawing were given heavier weight than those indicated by broken lines. One literature reference [24] was found subsequent to the Ottawa meeting and is not included in Figure 2.

In searching the literature a number of other references [25,26,27,28, 29,30,31,32,33,34,35,36] were found which gave data at temperatures below -250°C; these data were not included in the present survey, but the references are given for the sake of completeness.

Inspection of Figure 2 reveals that all data except those of three investigations [15,18,19] appear to indicate a conductivity at 0°C within about 2 percent of the deduced value for 0°C. At both lower and higher temperatures, the various data diverge markedly. It is estimated that the uncertainty of the values given in Table 2 is about 2 percent at 0°C, but increases to about 5 percent at -250°C and at 300°C. Because there is uncertainty as to the purity of the leads used in some of the investigations, the values in Table 2 are not considered to establish a best line for pure lead.

The National Bureau of Standards has recently issued a new lot of freezing point lead (Standard Sample 49e; the freezing point is 327.417°C ±0.005 degree). The same lead might also prove useful as a thermal conductivity standard. The Heat Transfer Section has obtained a sufficient quantity of this lead to fabricate a specimen for the NBS metals apparatus. We plan, as soon as the work load permits, to measure both the thermal conductivity and the electrical resistivity of this lead from about -160° to 250°C.

4. INCONEL 702

In order to obtain the highest possible accuracy in comparative thermal conductivity measurements, it is desirable to have a reference material having a thermal conductivity approximately similar to that of the test specimen materials. In view of the current importance of heat resistant alloys such as stainless steels and nickel-chromium alloys, a reference material of this type would appear to be quite useful. Powell and Tye [37] have suggested the use of "Nicloy G steel" as a thermal conductivity standard and have measured the thermal and electrical conductivity of this material (43.75% Fe, 36.5% Ni, 16.75% Cr) over the temperature range 0° to 800°C. In another recent paper [38], the same authors reported thermal and electrical conductivities for a number of similar nickel-chromium alloys. It can

be seen from the data given in these two papers that the thermal conductivity of these highly alloyed materials is relatively insensitive to minor changes in composition. Furthermore, there is good correlation between thermal and electrical conductivity.

A few years ago, the NBS Heat Transfer Section procured a stock of Inconel 702, which was chosen because of its excellent oxidation resistance at temperatures up to 1300°C. An NBS chemical analysis gave the following composition, in weight percent: Ni 79.3, Cr 17.0, Al 2.5, Ti 0.59, Fe 0.36, Si 0.19, Cu 0.14, Co 0.08, Mn 0.04, C 0.066, P 0.002, S 0.004. The Inconel 702 stock obtained by NBS was in a solution-annealed condition (i.e., had been held at 1080°C for one hour followed by rapid air cooling) when received from the suppliers. If held at temperatures between about 650° and 900°C, this alloy will become age-hardened due to precipitation of particles from the solid solution.

The thermal conductivity of specimens fabricated from this lot of Inconel 702 has been measured at NBS by three different methods, using five different apparatus. The results obtained have been used to deduce probable values for the thermal conductivity of this alloy in both the solution-annealed and the age-hardened condition. The values obtained are listed in Table 3. Above about 800°C, the thermal conductivity of this alloy is relatively independent of thermal history and a single column is given.

TABLE 3

THERMAL CONDUCTIVITY OF INCONEL 702

Temperature	Solution-annealed	Age-hardened
-150°C	0.099 w/cm C	-----
-100	.103	-----
0	.114	-----
100	.127	-----
200	.141	0.145 w/cm C
300	.158	.162
400	.176	.179
500	.195	.198
600	.216	.218
700	.237	.239
800	0.259	
900	.281	
1000	.301	
1100	.321	
1200	.340	

The thermal conductivity of solution-annealed Inconel 702 is shown plotted against temperature in the lower drawing of Figure 3. In the upper drawing, the smoothed results obtained in each of the several apparatus are shown as departures from the curve in the lower drawing. The solid curves refer to solution-annealed metal below 650°C; the broken curves refer to metal which had been age-hardened. The curve labeled M represents manually smoothed data from numerous runs on two specimens in two models of the NBS metals apparatus [5,6]. The curve labeled P corresponds to a quadratic equation of least-mean-squares fit to the data obtained in an early model of the NBS absolute cut-bar apparatus [55] on a specimen 2.54 cm in diameter and 3.81 cm long. The curve labeled A corresponds to the cubic equation of least-mean-squares fit to the data obtained in the NBS high temperature absolute cut-bar apparatus [57] on a specimen 2.54 cm in diameter and 6.59 cm long. The curve labeled A' corresponds to the cubic equation of least mean squares fit to the data obtained in the NBS high temperature absolute cut-bar apparatus on the same specimen as was used for curve A, which had become age-hardened in the apparatus at the higher temperatures of curve A. The specimen was not disturbed between these sets of tests. The curve labeled S corresponds to the linear equation of least-mean-squares fit to the data obtained in the NBS steam calorimeter apparatus on an age-hardened specimen 15.2 cm in diameter and 2.54 cm thick.

It can be seen in Figure 3 that all of the smoothed results obtained for the solution-annealed alloy to 800°C (M,P,A) are within plus or minus one percent of the values given in column two of Table 3. The thermal conductivity values obtained in the high temperature absolute cut-bar apparatus for the age-hardened alloy (curve A') should be quite good relative to values obtained in the same apparatus on the same specimen in the solution-annealed condition. The divergence of curve S from curve A' is believed to be due to inaccuracies in the data obtained using the steam calorimeter apparatus (i.e., curve S is believed to be in error). We plan to measure the thermal conductivity of an age-hardened specimen in the NBS metals apparatus.

Electrical resistivity measurements have been made on solution-annealed Inconel 702 from -195° to 300°C. Powell and Tye [38] found that the thermal conductivity of a group of nickel-chromium alloys could be predicted within 5 percent or better over the temperature range from 50° to 800°C by means of the equation

$$k' = 2.2\times10^{-8}(T/\rho) + 0.060 \qquad (1)$$

where k' is the predicted thermal conductivity (w/cm C), T is the absolute temperature (°K), and ρ is the electrical conductivity (ohm-cm). Using the electrical resistivity values measured, equation (1) was used to calculate

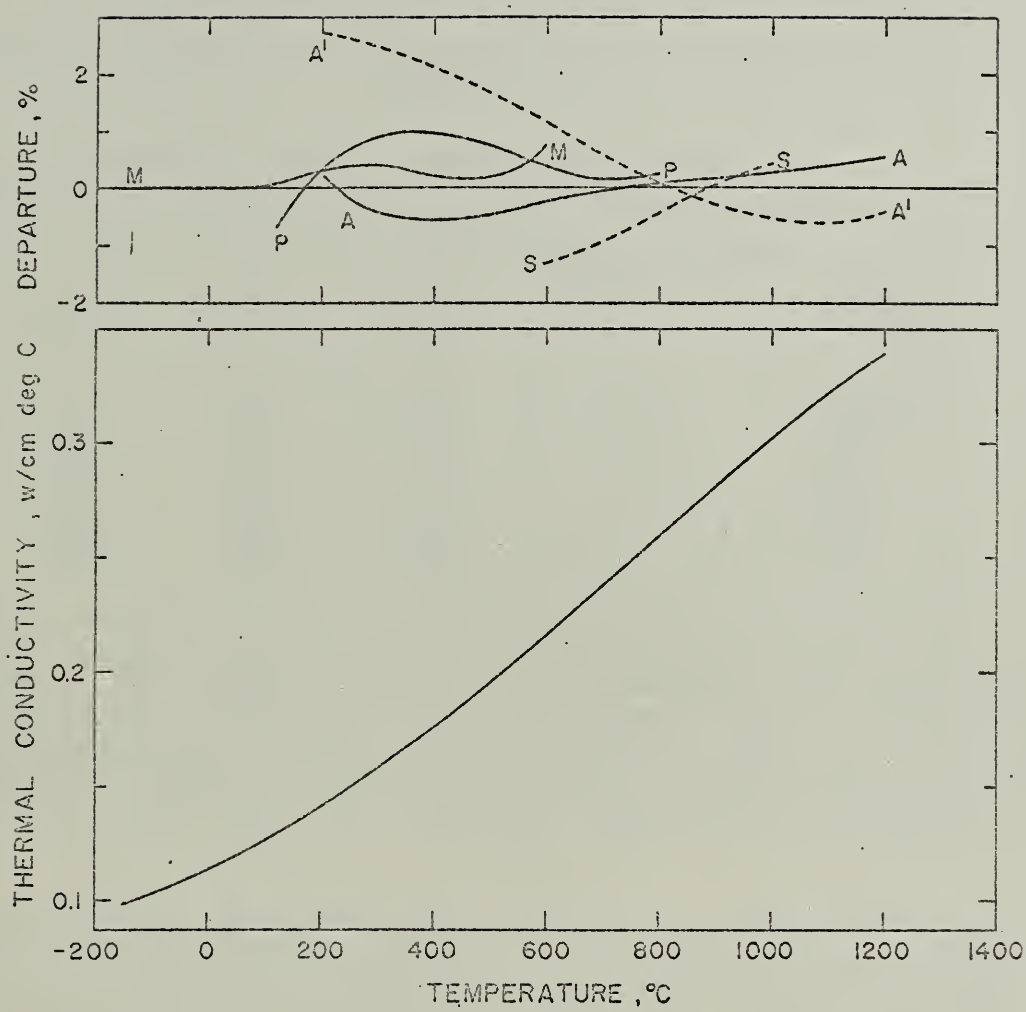

FIGURE 3. THERMAL CONDUCTIVITY OF INCONEL 702 IN THE SOLUTION-ANNEALED CONDITION (M,P,A) AND IN THE AGE-HARDENED CONDITION (A',S). THE UPPER DRAWING SHOWS DEPARTURES OF THE SMOOTHED RESULTS OBTAINED IN SEVERAL APPARATUS

M - Metals apparatus
P - Prototype absolute cut-bar apparatus
A,A' - High temperature absolute cut-bar apparatus
S - Steam calorimeter apparatus

thermal conductivity values for solution-annealed Inconel 702. Table 4 presents the measured values for electrical resistivity, ρ, and thermal conductivity, k, the calculated thermal conductivity values, k', and the percent departure, 100(k'-k)/k, of the calculated values from the measured values, and the Lorentz function kρ/T, from -150°C to 300°C.

TABLE 4

CORRELATION BETWEEN THERMAL CONDUCTIVITY AND ELECTRICAL RESISTIVITY OF SOLUTION ANNEALED INCONEL 702

t °C	ρ μΩ-cm	k w/cm C	k' w/cm C	100(k-k')/k %	kρ/T v^2/deg^2
-150	122.7	0.099	0.080	-19	9.86×10^{-8}
-100	123.9	.103	.091	-12	7.37
0	125.8	.114	.108	-5	5.25
100	127.0	.127	.125	-2	4.32
200	127.8	.141	.141	0	3.81
300	128.5	.158	.158	0	3.54

In the temperature range for which it was intended, Powell and Tye's equation is seen to hold very well. At lower temperatures, the calculated values depart significantly from the observed values. This is not surprising, especially in view of the very high values obtained for the Lorentz function (the theoretical value for electronic conduction is 2.443×10^{-8} volt^2/deg^{2}K).

We intend to measure the electrical resistivity of Inconel 702 up to at least 1200°C, with particular attention to the effects of heat treatment. Samples of this alloy (cut from the same plate as were the NBS specimens) have been sent to Dr. R. W. Powell of the National Physical Laboratory (Teddington, Middlesex, England), to Dr. M. J. Laubitz of the National Research Council (Ottawa, Ontario, Canada), to Dr.-Ing. Karl-Heinz Bode of the Physikalisch-Technische Bundesanstalt (Braunschweig, West Germany), and to several laboratories in the United States. In conjunction with thermal conductivity measurements, many of these laboratories will also make electrical resistivity determinations.

5. PYROCERAM CODE 9606

Measurements have been made of the thermal conductivity of a micro-crystalline glass (Pyroceram 9606, product of Corning Glass Works, Corning, New York). Microcrystalline glass is first formed as a homogeneous glass (incorporating a nucleating agent) which is transparent, so that any defects can be readily detected visually. By suitable heat treatment, the glass is later converted (by Corning) to a polycrystalline solid, which is opaque as a result of the large number of very small crystals. The properties of such materials have been described in the literature [39,40].

Measurements have been made in the NBS high temperature absolute cut-bar apparatus [57] on a specimen in the form of a cylinder 2.540 cm in diameter and 1.269 cm in length. The density of the specimen material was 2.601 g/cm^3 (value supplied by Corning). The thermal resistivity of Pyroceram Code 9606 was found to increase linearly from 200° to 1000°C, and can be represented by the equation

$$w = 1/k = 26.7 + 9.7\left(\frac{T}{1000}\right) \qquad (2)$$

with a standard deviation of less than one percent, where w is thermal resistivity (cm deg C/watt), k is thermal conductivity (watt/cm deg C), and T is temperature (°C). Thermal conductivity and resistivity values calculated from equation (2) are shown in Figure 4 and are listed in Table 5.

TABLE 5

THERMAL CONDUCTIVITY AND RESISTIVITY OF PYROCERAM CODE 9606

T °C	k w/cm C	w cm C/w
200	0.0350	28.6
400	.0327	30.6
600	.0308	32.5
800	.0290	34.5
1000	.0275	36.4

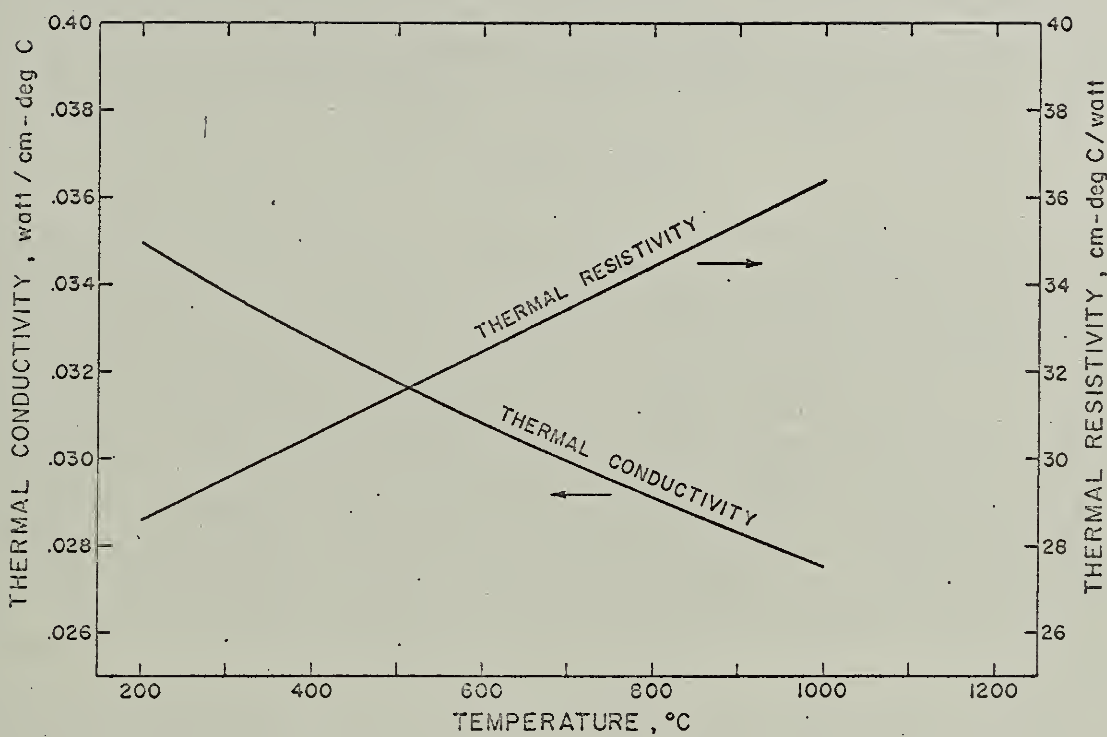

FIGURE 4. THERMAL CONDUCTIVITY AND THERMAL RESISTIVITY OF PYROCERAM CODE 9606 MICROCRYSTALLINE GLASS

The specimen was held at 1000°C for about 275 hours; data taken after this extended heat treatment were in substantial agreement with those taken before the specimen was held at 1000°C, thus indicating that no appreciable change in the thermal conductivity of Pyroceram 9606 occurred as a result of this heat treatment.

Measurements on Pyroceram 9606 have also been made in the NBS metals apparatus over the range 100° to 650°C. The data have not been completely analyzed, but appear to be in good agreement with the values obtained in the high temperature absolute cut-bar apparatus. Thermal diffusivity values for Pyroceram Code 9606 have been reported by Plummer, Campbell, and Comstock [41]. Thermal diffusivity measurements are also being made by H. W. Flieger and D. C. Ginnings of the NBS Heat Measurements Section.

6. PYREX GLASS

The thermal conductivity of Pyrex glass has been measured by numerous investigators, and this material has been used for calibration or checking of thermal conductivity apparatus. The NBS Heat Transfer Section has measured the thermal conductivity of Pyrex Code 7740 in the NBS guarded hot plate apparatus and in an early model of the NBS absolute cut-bar apparatus. This glass was found to have a density of 2.2258 g/cm^3 at 24.3°C. For further identification, the refractive index was measured at 23°C and found to be 1.47257±0.00003 for the D lines of sodium (5893Å). In the course of determining the index of refraction, it was noted that the glass prism (prepared from polished Pyrex plate) was somewhat strained. After annealing, the index of refraction was found to have decreased to a value of 1.47211 ±0.00003. Thermal conductivity specimens were not annealed prior to measurement.

The NBS results were used, in conjunction with literature values and several unpublished data available to us from other laboratories, to deduce probable values for the thermal conductivity of Pyrex glass. The values arrived at are given in Table 6 and are shown plotted in the lower curve of Figure 5.

The various determinations [42,44,45,47,48 and 49,50,51,52,53,54,55] of the thermal conductivity of Pyrex glass are shown in the upper drawing of Figure 5 as percent departures from the smooth curve in the lower drawing. Two references [43,46] were excluded as being outside the range of the drawing. One reference [41] was received too late to be included. It is very doubtful that the scatter seen would be due to differences in chemical composition [41,56]. Differences in the microstructure could be responsible [41] for some of the observed differences, but most of these must be

attributed to experimental errors. It is estimated that the derived values are given in Table 6 are uncertain by not more than 5 percent below 300°C. Above this temperature, radiation effects cause the thermal conductivity to become a somewhat ambiguous property. We plan to make additional measurements of the thermal conductivity of Pyrex Code 7740 glass.

TABLE 6

DEDUCED VALUES FOR THE THERMAL CONDUCTIVITY OF PYREX GLASS

Temperature °C	Thermal Conductivity watt/cm deg C
0	0.0111
50	.0116
100	.0122
150	.0127
200	.0133
250	.0138
300	.0143
400	(0.0154)
500	(0.0165)

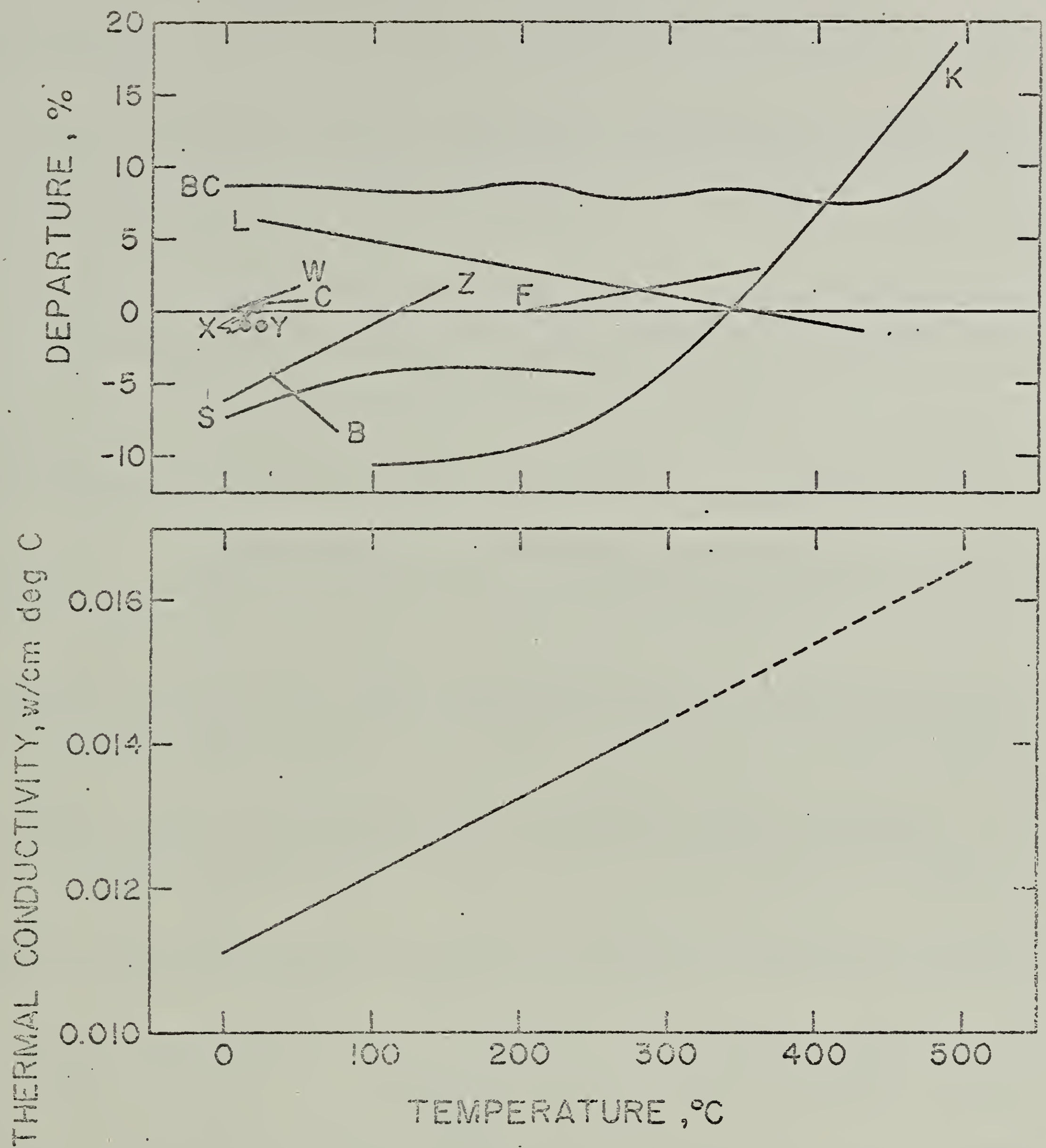

FIGURE 5. DEDUCED VALUES FOR THE THERMAL CONDUCTIVITY OF PYREX GLASS. THE UPPER DRAWING SHOWS DEPARTURES OF DATA OF VARIOUS INVESTIGATORS FROM THE DEDUCED VALUES

B Bridgman (42)
S Stephens (44)
BC Birch & Clark (45)
L Lucks, Deem & Wood (47)
K Kingery (48 & 49)
C Challoner, Gundry & Powell (50)
X Laboratory X (51)
Y Laboratory Y (52)
Z Laboratory Z (53)
W Watson (54)
F Flynn (55)

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